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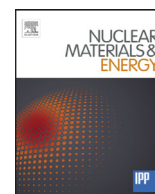
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Deuterium retention in W and W-Re alloy irradiated with high energy Fe and W ions: Effects of irradiation temperature



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ABSTRACT

Neutron irradiation to W induces defects acting as traps against hydrogen isotopes and transmutation elements such as Re and Os. To investigate synergetic effects on radiation-induced defects and Re, deuterium (D) retention in W and W–5% Re samples were examined after irradiation with 6.4 MeV Fe ions at 523–1273 K followed by exposure to D₂ gas at 673 K. The value of D retention in W–5% Re was lower than that in W by orders of magnitude after the irradiation at high temperatures (≥ 1073 K), while no significant effects of Re addition was observed after irradiation at 523 K. Irradiation with 20 MeV W ions at room temperature followed by exposure to D plasma at 443–743 K also resulted in small difference in D retention between W and W–5% Re samples. The results of positron lifetime measurements showed that the reduced D retention by Re observed after high temperature irradiation was due to suppression of formation of vacancy-type defects (monovacancies and vacancy clusters) by Re.

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1. Introduction

Due to its favorable physical properties, such as low erosion yield and high melting point, tungsten (W) has been recognized as a candidate material for plasma-facing high heat-flux structures in future fusion reactors. As plasma-facing material in fusion reactors, W will be exposed to intensive fluxes of energetic deuterium (D) and tritium (T) particles, as well as 14 MeV neutrons (n) from the D–T fusion reaction. Irradiation of W with fusion neutrons creates transmutation elements such as rhenium (Re) and osmium (Os). There have been a number of studies trying to predict the amount of Re accumulated in W during fusion power plant operation [1–4]. The most recent forecast by Gilbert and Sublet [4] gives 0.18 at.% of Re for 14 years of ITER operation and 3.8 at.% of Re for 5 years of DEMO reactor operation. Neutron irradiation generates also displacements in the bulk of W and creates defects at which hydrogen isotopes can be trapped. It has been reported that the

neutron irradiation of W at ~ 323 K to 0.025–0.3 displacements per atom (dpa) and subsequent exposure to D plasma at 473 and 773 K result in trapping of D up to 0.1–1 at.% [5–7]. However, the influence of transmutation elements and high temperature irradiation has not been examined.

As reviewed in [8–10], the influence of neutron-produced defects on the hydrogen isotope inventory has been investigated by simulating displacement damage in W by irradiation with high energy ions and then loading the damaged zone with D. Ogorodnikova et al. [11] have studied the D trapping in W after irradiation of 20 MeV W ions at elevated temperatures followed by plasma exposure and compared the results with those obtained with post-irradiation annealing after the irradiation at room temperature. They reported that irradiation of W at 500, 650 and 950 K reduced the concentration of trapped D by factors of ~ 1.5 , ~ 2.2 and ~ 2.8 , respectively, compared to W irradiated at 300 K due to dynamic annealing under the irradiation. The extent of defect recovery by the dynamic annealing was slightly larger than that observed after post-irradiation annealing at comparable temperatures. Regarding the effects of transmutation elements,

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Tyburska-Püschel and Alimov [12] have irradiated W and W–3% Re samples with 20 MeV W ions at room temperature and then exposed to low-energy D plasma at elevated temperatures to the fluence as high as 1×10^{26} D/m² (W) and 3×10^{26} D/m² (W–3% Re). They reported that the concentration of D trapped at defects in W–3% Re was lower than that in W by orders-of-magnitude at exposure temperatures of 750 K [12].

In this study, the synergetic effects of Re addition and high temperature irradiation were examined by irradiating W and W–5% Re samples with 6.4 MeV Fe ions at 523–1273 K. The irradiated samples were exposed to D₂ gas at 673 K, and the concentration of trapped D (C_{D-trap}) was measured using nuclear reaction analysis (NRA) and thermal desorption spectrometry (TDS). The effects of post-irradiation annealing and D plasma exposure at elevated temperatures after irradiation at low temperature were also examined. In this case, W and W–5% Re samples irradiated with 6.4 MeV Fe ions at 523 K and those damaged with 20 MeV W ions at room temperature were used. The former set of samples were annealed in vacuum at 1073 K and then exposed to D₂ gas at 673 K. The latter set of samples were exposed to low-energy, high flux D plasma, as performed by Tyburska-Püschel and Alimov [12], at 443–743 K to the fluence of 3×10^{25} D/m²; the D fluence in this study was lower than that in [12] by a factor of 10. Results obtained showed that the C_{D-trap} in W–5% Re is significantly lower than that in W after irradiation at high temperatures, while the difference between W–5% Re and W is small after low temperature irradiation and that followed by post-irradiation annealing or D plasma exposure at elevated temperatures. The mechanisms underlying the reduced C_{D-trap} after high temperature irradiation is discussed.

2. Experimental

Two W-based materials were used in this study: (i) 1 mm thick warm-rolled W plate of 99.95 mass% purity from Goodfellow Cambridge Ltd, England, and (ii) 0.5 mm thick warm-rolled W–5% Re alloy plate of 99.99 mass% purity delivered by A.L.M.T. Co., Japan. After cutting of the plates into samples 10×10 mm² in size, the samples were mechanically polished, cleaned in an acetone ultrasonic bath, and annealed in vacuum at 1223 K for 1 h to relieve stresses occurred in the polishing process.

The W and W–5% Re samples were irradiated with 6.4 MeV Fe ions at temperatures of 523, 773, 1073, and 1273 K to the same fluence of 3.2×10^{18} Fe/m² in the accelerator DuET, Kyoto University. The sample temperature was set to the above-mentioned irradiation temperature before starting the irradiation by a heater placed in the back of a sample holder. After starting the irradiation, the power supplied to the heater was gradually reduced to keep the sample temperature constant under beam heating. An optical pyrometer was used to monitor the temperature of irradiated surface of the sample. The damage profile was calculated using the program SRIM 2008.03 [13], “full cascade option”, with a displacement threshold energy of $E_d = 90$ eV. The calculation indicated that the near-surface layer of the samples was damaged to 0.5 displacements per atom (dpa) at the damage peak situated at a depth of 1.2 μ m. Some of the samples irradiated at 523 K were annealed in a vacuum at 1073 K for 1 h (that comparable with the time period of the Fe ion irradiation) to examine the effects of post-irradiation annealing.

All the samples damaged with 6.4 MeV Fe ions were exposed to D₂ gas at temperature of 673 K and pressure of 100 kPa for 10 h. For exposure to D₂ gas, the samples were placed inside the quartz tube connected to a high-vacuum pumping system and heated in a vacuum (10^{-5} Pa) with the use of an external ohmic heater. The temperature was monitored using a type K thermocouple located near the samples inside the tube. As the sample temperature reached 673 K, a valve between the tube and the pumping system

was closed and the tube was filled with D₂ gas of 0.1 MPa. After reaching required exposure duration (10 h), D₂ gas evacuation and sample cooling started simultaneously. D₂ gas was evacuated in several seconds, while the sample was cooled down in several minutes.

A part of W and W–5% Re samples were subjected to irradiation of 20 MeV W ions at room temperature at the Max-Planck-Institut für Plasmaphysik to a fluence of 8×10^{17} W m^{–2}. The near-surface layer of the samples was damaged to 0.5 dpa at the damage peak situated at a depth of 1.4 μ m. The samples irradiated with 20 MeV W ions were exposed to low-energy (76 eV/ion), high flux ($\approx 10^{22}$ D/m² s) D plasma at 443–743 K to the fluence of 3×10^{25} D/m² in the linear plasma generator at the International Fusion Energy Research Center (IFERC), Japan Atomic Energy Agency (JAEA). The conditions of W ion irradiation and D plasma exposure were similar to those in [12] except that the fluence of D in this study was lower than that in [12] by a factor of 10.

The deuterium depth profiles in the damaged samples were determined by nuclear reaction analysis (NRA) at the Max-Planck-Institut für Plasmaphysik. The D(³He, p)⁴He reaction was utilized, and both the α particles and protons were analyzed. The α -spectrum was transformed into a D depth profile at depths up to ~ 0.5 μ m using the program SIMNRA [14]. To determine the D concentration at larger depths, the energy of the analyzing beam of ³He ions was varied from 0.8 to 4.0 MeV. The proton yields measured at different ³He ion energies allow D depth profiles to be measured to depths of up to 7 μ m [15].

After the NRA measurements, W and W–5% Re samples irradiated with Fe ions at 1073 K were analyzed by thermal desorption spectrometry (TDS) at the IFERC, JAEA. A ceramic heater was used to heat the samples at a ramp rate of 0.5 K/s and the sample temperature was raised to 1273 K. HD, D₂ and other molecules released during the TDS run were monitored by a quadrupole mass spectrometer (QMS). A standard D₂ leak with an inaccuracy smaller than 10% was employed to calibrate the QMS.

The W and W–5% Re samples irradiated with 6.4 MeV Fe ions at 1273 K were subjected to positron lifetime measurement in the International Research Center for Nuclear Materials Science, the Institute for Materials Research, Tohoku University. A positron source (²²Na) sealed with Kapton films of ~ 1 MBq was used, and a total of 5×10^6 events were accumulated for each measurement using a conventional positron lifetime system with BaF₂ scintillators. The lifetime spectrum was analyzed using the PALSfit software.

3. Results

Fig. 1 shows depth profiles of D in the W and W–5% Re samples irradiated with 6.4 MeV Fe ions at various temperatures and then exposed to D₂ gas at 673 K. The concentration of D was high in the near-surface region up to the depth of ~ 2 μ m, indicating the trapping of D at radiation-induced defects. As the Fe ion irradiation temperature, T_{Fe-irr} , increased, the concentration of trapped D, C_{D-trap} , decreased.

The correlation between C_{D-trap} at the damage peak and T_{Fe-irr} is shown in Fig. 2. The value of C_{D-trap} in W–5% Re was comparable with that in W after Fe ion irradiation at 523 K. The reduction in C_{D-trap} with increase in T_{Fe-irr} was observed for both materials, but the extent of reduction of W–5% Re was significantly larger than that of W. In other words, C_{D-trap} in W–5% Re at $T_{Fe-irr} = 1273$ K was 1/180 of that at $T_{Fe-irr} = 523$ K, while C_{D-trap} in W at $T_{Fe-irr} = 1273$ K was smaller than that at $T_{Fe-irr} = 523$ K by a factor of 3. The extent of difference in C_{D-trap} in W between $T_{Fe-irr} = 523$ and 1273 K (a factor of 3) was comparable with that observed in [11]. This figure also shows the values of C_{D-trap} in the W and W–5% Re samples subjected to the post-irradiation annealing at 1073 K after Fe ion irradiation at 523 K. The value of C_{D-trap} in W samples after the ir-

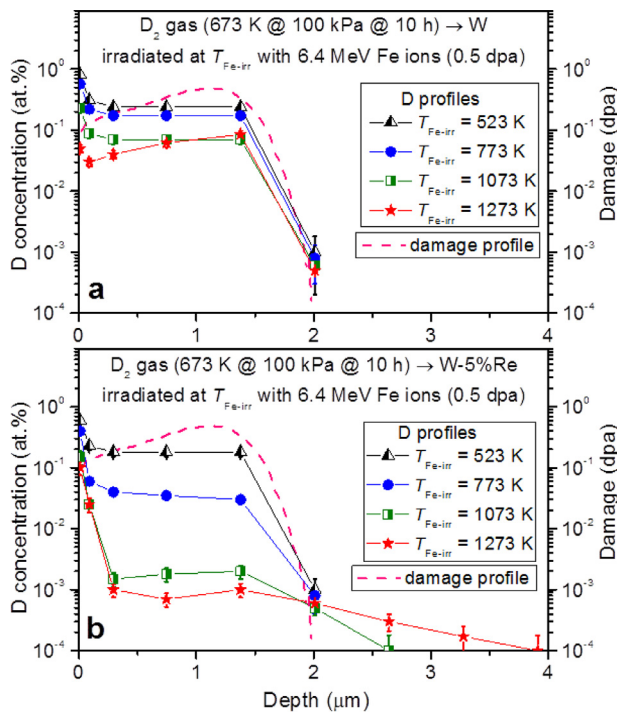


Fig. 1. Depth profiles of D retained in W (a) and W-5% Re (b) irradiated at various temperatures with 6.4 MeV Fe ions to the peak damaged level of 0.5 dpa and then exposed to D₂ gas at temperature of 673 K and pressure of 100 kPa for 10 h. Temperatures of irradiation with 6.4 MeV Fe ions, T_{Fe-irr} , are indicated in the legends. In both panels, a calculated damage depth profile is also shown.

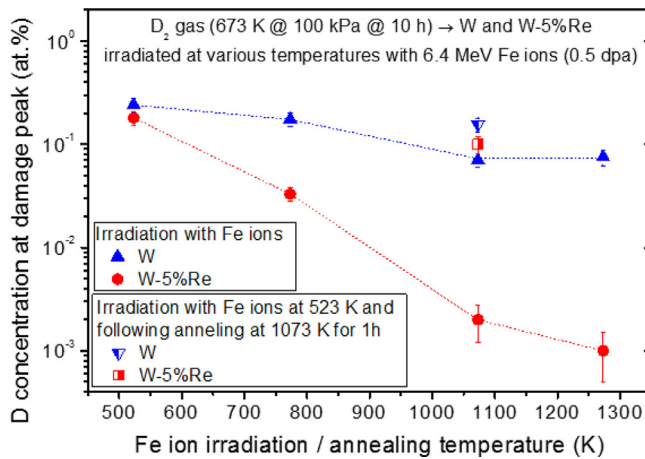


Fig. 2. Concentration of deuterium at a depth of the damage peak in W and W-5% Re irradiated at various temperatures with 6.4 MeV Fe ions to the peak damage level of 0.5 dpa and then exposed to D₂ gas at a temperature of 673 K and a pressure of 100 kPa for 10 h, as a function of Fe ion irradiation temperature. Additionally, the D concentration at the damage peak in W and W-5% Re, initially irradiated at 523 K with 6.4 MeV Fe ions to the peak damage level of 0.5 dpa and then annealed in vacuum at 1073 K for 1 h, after D₂ gas exposure under the same exposure conditions is shown.

radiation at 1073 K was slightly lower (by a factor of 2) than that after the post-irradiation annealing at 1073 K, as reported in [11]. Far larger difference between dynamic annealing during the irradiation and post-irradiation annealing was observed for W-5% Re samples; C_{D-trap} after the irradiation at 1073 K was lower than that after the post-irradiation annealing by a factor of 50.

Fig. 3 shows TDS spectra of D released from the W and W-5% Re samples damaged with 6.4 MeV Fe ions at 1073 K. Deuterium was mainly desorbed as D₂ molecules, and the signal intensity for

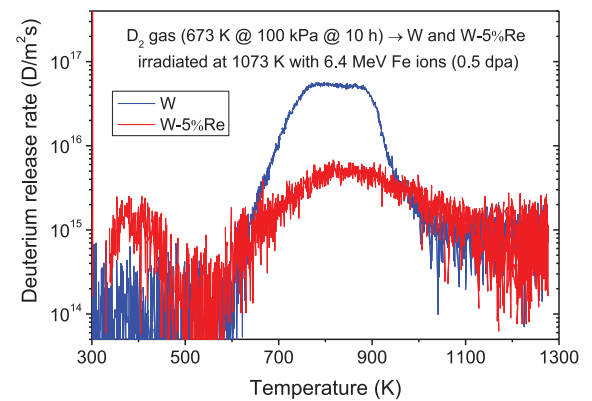


Fig. 3. Thermal desorption spectra of deuterium for W and W-5% Re irradiated at 1073 K with 6.4 MeV Fe ions to the peak damaged level of 0.5 dpa and then exposed to D₂ gas at a temperature of 673 K and a pressure of 100 kPa for 10 h.

Table 1

Results of positron lifetime measurements for W and W-5% Re samples before and after the irradiation with 6.4 MeV Fe ions at 1273 K to 0.5 dpa.

Sample	W (non-irradiated)	W (irradiated)	W-5% Re (non-irradiated)	W-5% Re (irradiated)
Positron lifetime (ps)	133.9 ± 0.5	125 ± 1, 470 ± 20 (ave. 168.0 ± 0.5)	137.7 ± 0.5	138.9 ± 0.5

HD, CH₃D, HDO and D₂O were lower than that of D₂ by an order of magnitude or more. A broad desorption peak at 700–950 K was observed for both samples. The W-5% Re sample showed a small additional peak at around 400 K. The values of D retention in the W sample measured using the NRA and TDS techniques were $(7 \pm 1) \times 10^{19}$ D/m² (NRA) and $(2 \pm 1) \times 10^{19}$ D/m² (TDS), whereas for the W-5% Re sample these values were $(6 \pm 2) \times 10^{18}$ D/m² (NRA) and $(4 \pm 2) \times 10^{18}$ D/m² (TDS). It should be noted that the TDS measurements were performed about 3 months after the NRA measurements, and hence desorption of D at weak traps was possible before the TDS measurements.

The results of positron lifetime measurements are summarized in Table 1. The values of positron lifetime in non-irradiated W and W-5% Re samples were 133.9 ± 0.5 and 137.7 ± 0.5 ps, respectively. According to Troev et al. [16], the positron lifetime in W matrix is 108 ps and that in a monovacancy is 200 ps. The values of positron lifetime observed for the non-irradiated W and W-5% Re samples appear to be average values determined by the matrix of W and intrinsic defects such as dislocations and vacancies induced during fabrication processes at relatively low concentrations. The irradiation of W with 6.4 MeV Fe ions to 0.5 dpa at 1273 K resulted in significant increase in positron lifetime. The lifetime spectrum was fitted well using two components, 125 ± 1 ps and 470 ± 20 ps; the average lifetime was 168.0 ± 0.5 ps. The value for the short-life component (125 ± 1 ps) is close to the positron lifetime in the non-irradiated samples, while that for long-life component (470 ± 20 ps) corresponds to relatively large vacancy clusters. Troev et al. [16] reported that the positron lifetime in 37V cluster (V is a monovacancy) was evaluated to be 437 ps. The W sample irradiated with Fe ions at 1273 K appears to contain vacancy clusters larger than 37V. In contrast, no significant increase in positron lifetime was observed for W-5% Re after the irradiation of Fe ions under the same conditions; the positron lifetime in the W-5% Re sample irradiated at 1273 K was evaluated to be 138.9 ± 0.5 ps. These observations suggest that Re significantly suppressed the growth of vacancy clusters. Tanno et al. [17] and Fukuda et al. [18,19] have observed microstructures of W and

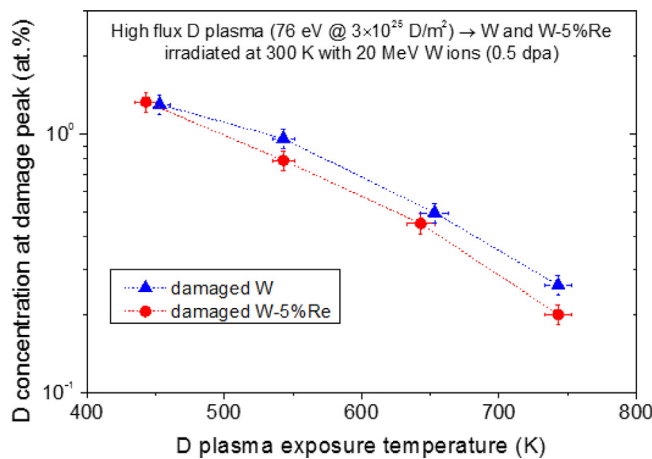


Fig. 4. Concentration of deuterium at a depth of the damage peak in W and W-5% Re irradiated at room temperature with 20 MeV W ions to the peak damage level of 0.5 dpa and then exposed to low energy, high flux D plasma at 443–743 K.

W-Re alloys with a transmission electron microscope after neutron irradiation at elevated temperatures. They reported that the density of voids in W-Re alloys was lower than that in W [17–19], while the former contains precipitates consisting of W and Re [17]. The results of positron lifetime measurements in this study were consistent with their observations. As previously mentioned, the positron lifetime in a monovacancy is 200 ps [16] and significantly longer than that in the matrix of W-5% Re. Negligible increase in positron lifetime after Fe ion irradiation at 1273 K indicates that Re suppressed also the formation of monovacancies.

Fig. 4 shows C_{D-trap} in the W and W-5% Re samples irradiated with 20 MeV W ions at room temperature and then exposed to low-energy, high-flux D plasma at 443–743 K as a function of exposure temperature, T_{exp} . The value of C_{D-trap} in both W and W-5% Re decreased with increase in T_{exp} . Although C_{D-trap} in W-5% Re systematically gave smaller values than that in W, the difference between W and W-5% Re in Fig. 4 was far smaller than that observed after Fe ion irradiation at elevated temperatures (Fig. 2); the difference between W and W-5% Re was just by a factor of 1.3 or less in Fig. 4, while C_{D-trap} in W-5% Re after Fe ion irradiation at 773 K was smaller than that in W by a factor of 5. Tyburska-Püschel and Alimov reported that C_{D-trap} in W-3% Re was smaller than that in W by two orders of magnitude at $T_{exp} = 760$ K (Fig. 3 in [12]). The extent of difference between W and W-5% Re shown in Fig. 4 was markedly small in comparison with the observations of Tyburska-Püschel and Alimov [12].

4. Discussion

The depth profiles given in Fig. 1 shows that C_{D-trap} is almost uniform within the damaged zone except the near-surface regions below a depth of ~ 100 nm. These observations suggest that traps were induced almost uniformly in the damaged zone under the present conditions. It was mentioned above that the W and W-5% Re samples were irradiated at various temperatures with 6.4 MeV Fe ions to a fluence of 3.2×10^{18} Fe/m². As this takes place, a concentration of Fe atoms at the peak of the Fe ion depth distribution (at a depth of ~ 1.2 μ m), as calculated using the program SRIM 2008.03 [13], is 4.7×10^{24} Fe/m³. Thus, the Fe concentration in W and W-5% Re is below $10^{-2}\%$. The concentration of Re (5%) and the values of C_{D-trap} in W and W-5% Re samples irradiated with Fe ions at 523 and 773 K are higher than the concentration of Fe by orders of magnitude. It is appropriate to consider that the influence of Fe atoms on D trapping in these samples was negligibly small and the trapping effects were dominated by defects induced

by Fe ion irradiation. In contrast, the values of C_{D-trap} in the W-5% Re samples irradiated at 1073 K and 1273 K were 10^{-3} at.% and lower than the above-mentioned value of Fe concentration. However, detailed discussion on the trapping effects of Fe is difficult due to possible diffusion of Fe at high temperatures. As described above, a small desorption peak was observed at around 400 K for W-5% Re sample. Because no clear peak was observed in this temperature range for Fe-irradiated W, it is obvious that this peak does not correspond to D trapped at Fe atoms. Oya et al. [20] have reported that D trapped at dislocation loops forms desorption peak in this temperature range.

The concentration of trapped D, C_{D-trap} , is expressed as follows:

$$C_{D-trap} = \sum_i C_{t,i} \theta_{t,i}, \quad (1)$$

where $C_{t,i}$ is the concentration of accommodation site i for D in defects, and $\theta_{t,i}$ is the fraction of the accommodation site i occupied by a D atom ($\theta_{t,i} \leq 1$). It should be noted that $C_{t,i}$ is not the concentration of defects, because a defect can trap multiple D atoms. For example, the first-principle calculations showed that a monovacancy in W traps 6 or more D atoms in different positions, and the binding energy between monovacancy and D atom in an interstitial site varies with the number of trapped D atoms [21–24]. In other words, a monovacancy in W provides 6 or more accommodation sites for D atoms with different values of binding energy. Similar trapping of multiple D atoms is possible also for other types of defects. It is clear that the concentration of accommodation sites is proportional to that of radiation-induced defects. Because only one D atom can be embedded in each accommodation site, the occupation probability $\theta_{t,i}$ at T_{exp} under local trapping-detrapping equilibrium is expressed as [25]

$$\frac{\theta_{t,i}}{1 - \theta_{t,i}} = \frac{\theta_L}{1 - \theta_L} \exp\left(\frac{E_{bin,i}}{kT_{exp}}\right), \quad (2)$$

where θ_L is the fraction of occupied interstitial sites, $E_{bin,i}$ is the binding energy between site i and D atom in an interstitial site and k is the Boltzmann constant. Under exposure to D₂ gas, θ_L is determined by the solubility of D, the pressure of D₂ gas and temperature. Benamati [26] et al. have measured the permeability of H₂ and D₂ gases through W and W-5% Re and reported that the values of permeability for W and W-5% Re were comparable with each other. It is therefore plausible that the solubility of D in W and W-5% Re are also comparable with each other, as well as the diffusivity.

The data shown in Figs. 1 and 2 were acquired after Fe ion irradiation at different temperatures followed by the exposure to D₂ gas under the same conditions (673 K and 0.1 MPa). Therefore, the values of θ_L and T_{exp} are the same for all data shown in these figures. The solubility of D in W-5% Re is comparable with that in W, as mentioned above. The TDS spectra of D released from W and W-5% Re samples irradiated at 1073 K (Fig. 3) gave the main peaks in the same temperature range, indicating that the values of E_{bin} are also comparable between W and W-5% Re. Therefore, solely the difference in the concentration of accommodation sites, $C_{t,i}$, can explain the significantly lower C_{D-trap} in W-5% Re than in W after Fe ion irradiation at high temperatures. In other words, lower C_{D-trap} was due to lower concentration of radiation-induced defects acting as trapping sites. The results of positron lifetime measurements summarized in Table 1 clearly indicates that the concentration of vacancy-type defects (monovacancies and vacancy clusters) in W-5% Re was significantly lower than that in W after the irradiation with Fe ions at 1273 K. It was therefore concluded that vacancy-type defects played dominant roles in D trapping after high temperature irradiation and the significantly reduced C_{D-trap} observed for W-5% Re was due to suppressed formation of vacancy-type defects during high temperature irradiation.

According to Suzudo et al. [27], Re atom in W forms mixed dumbbell with interstitial W atom. The rotation energy barrier of the W-Re dumbbell is so low that Re interstitials have 3-dimensional motion instead of the 1-dimensional motion of W self-interstitial atoms, and interstitials with 3-dimensional motion have a greater probability of reaching vacancy sites for recombination [27]. The mechanism proposed by Suzudo et al. [27] are readily applicable also for the case of this study.

The marked reduction in C_{D-trap} observed for W-5% Re after Fe ion irradiation at 773–1273 K was not reproduced by neither of post-irradiation annealing at 1073 K after Fe ion irradiation at 523 K (Fig. 2) nor exposure to the plasma at elevated temperatures (up to 743 K) after W ion irradiation at room temperature (Fig. 4). These observations suggest that dynamic annealing under irradiation plays a critical role in the reduction of concentrations of vacancy-type defects by Re addition. Further investigation is necessary to understand the detailed mechanisms underlying the enhanced recovery of vacancy-type defects by Re addition under dynamic annealing.

As mentioned in Sec. 3, the difference in C_{D-trap} between W-5% Re and W after high temperature plasma exposure observed in this study was far smaller than that reported in [12]. The fluence of D in the plasma exposure in [12] was higher than that in this study by a factor of 10, and consequently exposure time was also longer. However, the significant difference between this study and [12] cannot be attributed to the effects of longer annealing at T_{exp} . As reported in [28,29], the activation energy for vacancy migration in W is 1.7 eV. The number of jumps that vacancy can make during the plasma exposure at 760 K to the fluence of 3×10^{26} D/m² at the flux of $\approx 10^{22}$ D/m² s (30 ks exposure) is orders-of-magnitude smaller than that during the post-irradiation annealing at 1073 K for 1 h. The small effects of the post-irradiation annealing shown in Fig. 2 indicates that significant recovery of vacancy-type defects is impossible during the plasma exposure for 30 ks at 760 K. Therefore, the large reduction in C_{D-trap} observed by Tyburska-Püschel and Alimov [12] is, in our opinion, due to synergetic effects of presence of Re and stress induced by implantation of D to high fluence. It is plausible the interactions of D-induced defects, W-ion-induced defects and Re atoms occurred in the study of Tyburska-Püschel and Alimov [12], and the modified microstructure thus developed resulted in the large drop in C_{D-trap} after the plasma exposure at elevated temperatures. However, further investigation is necessary for full clarification of underlying mechanisms. The effects of implanted D can extend only to a limited depth of materials. The data given in Figs. 1–3 of this paper are free from those effects induced by D implantation. Therefore, the data in this study show more appropriately the effects of Re on hydrogen isotope trapping in the bulk of irradiated W materials, while those given by Tyburska-Püschel and Alimov [12] show Re effects in the near-surface region of plasma-facing components.

5. Conclusions

- (1) The concentration of radiation-induced traps in W-5% Re was orders-of-magnitude lower than that in W after irradiation of 6.4 MeV Fe ions at high temperatures (≥ 1073 K), while no significant effect of Re was observed after irradiation at 523 K and irradiation of 20 MeV W ions at room temperature.
- (2) The effects of post-irradiation annealing in vacuum at 1073 K after Fe ion irradiation at 523 K and that at 443–743 K during D plasma exposure after W ion irradiation at room temperature were small compared with the above-mentioned effects of high temperature irradiations of Fe ions.

- (3) The positron lifetime measurements showed that the presence of Re reduced the concentrations of vacancy-type defects during the high temperature irradiation.
- (4) The significant reduction in D retention by Re addition after the high temperature irradiation was ascribed to the lower concentration of vacancy-type defects acting as traps against D.
- (5) Re concentration and irradiation temperature are key factors in evaluating neutron irradiation effects on hydrogen isotope retention in W materials using heavy ion irradiation.

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References

- [1] T. Noda, M. Fujita, M. Okada, J. Nucl. Mater. 258–263 (1998) 934–939.
- [2] G.A. Cottrell, J. Nucl. Mater. 334 (2004) 166–168.
- [3] G.A. Cottrell, R. Pampin, N.P. Taylor, Fusion Sci. Technol. 50 (2006) 89–98.
- [4] M.R. Gilbert, J.-Ch. Sublet, Nucl. Fusion 51 (2011) 043005.
- [5] Y. Hatano, M. Shimada, T. Otsuka, Y. Oya, V.Kh. Alimov, M. Hara, J. Shi, M. Kobayashi, T. Oda, G. Cao, K. Okuno, T. Tanaka, K. Sugiyama, J. Roth, B. Tyburska-Püschel, J. Dorner, N. Yoshida, N. Futagami, H. Watanabe, M. Hatakeyama, H. Kurishita, M. Sokolov, Y. Kato, Nucl. Fusion 53 (2013) 073006.
- [6] Y. Hatano, M. Shimada, V.Kh. Alimov, J. Shi, M. Hara, T. Nozaki, Y. Oya, M. Kobayashi, K. Okuno, T. Oda, G. Cao, N. Yoshida, N. Futagami, K. Sugiyama, J. Roth, B. Tyburska-Püschel, J. Dorner, I. Takagi, M. Hatakeyama, H. Kurishita, M.A. Sokolov, J. Nucl. Mater. 438 (2013) S114–S119.
- [7] M. Shimada, G. Cao, T. Otsuka, M. Hara, M. Kobayashi, Y. Oya, Y. Hatano, Nucl. Fusion 55 (2015) 013008.
- [8] D.G. Whyte, J. Nucl. Mater. 390–391 (2009) 911–913.
- [9] O.V. Ogorodnikova, B. Tyburska, V. Kh. Alimov, K. Ertl, J. Nucl. Mater. 415 (2011) S661–S666.
- [10] J. Roth, K. Schmid, Phys. Scr. T145 (2011) 014031.
- [11] O.V. Ogorodnikova, Yu. Gasparyan, V. Efimov, Ł. Ciupiński, J. Grzonka, J. Nucl. Mater. 451 (2014) 379–386.
- [12] B. Tyburska-Püschel, V.Kh. Alimov, Nucl. Fusion 53 (2013) 123021.
- [13] J.F. Ziegler, SRIM – The Stopping and Range of Ions in Matter: ver. SRIM-2008.3, <http://srim.org>.
- [14] M. Mayer, SIMNRA User's Guide Tech. Rep. IPP 9/113, Max-Planck-Institut für Plasmaphysik, Garching, 1997.
- [15] V.Kh. Alimov, M. Mayer, J. Roth, Nucl. Instrum. Meth. B 234 (2005) 169–175.
- [16] T. Troev, E. Popov, P. Staikov, N. Nankov, T. Yoshiie, Nucl. Instrum. Methods Phys. Res. B 267 (2009) 535–541.
- [17] T. Tanno, A. Hasegawa, M. Fujiwara, J.-C. He, S. Nogami, M. Satou, T. Shishido, K. Abe, Mater. Trans. 49 (2008) 2259–2264.
- [18] M. Fukuda, T. Tanno, S. Nogami, A. Hasegawa, Mater. Trans. 53 (2012) 2145–2150.
- [19] M. Fukuda, K. Yabuuchi, S. Nogami, A. Hasegawa, T. Tanaka, J. Nucl. Mater. 445 (2014) 460–463.
- [20] Y. Oya, M. Kobayashi, T. Fujishima, K. Kawasaki, Y. Miyahara, N. Yoshida, Y. Hatano, N. Ashikawa, A. Sagara, K. Okuno, Mater. Trans. 54 (2013) 430–436.
- [21] D. Kato, H. Iwakiri, K. Morishita, J. Plasma Fusion Res. Ser. 8 (2009) 404–407.
- [22] Y.-L. Liu, Y. Zhang, H.-B. Zhou, G.-H. Lu, F. Liu, G.-N. Luo, Phys. Rev. B 82 (2010) 172103.
- [23] K. Heinola, T. Ahlgren, K. Nordlund, J. Keinonen, Phys. Rev. B 82 (2010) 09410.
- [24] K. Ohsawa, J. Goto, M. Yamakami, M. Yamaguchi, M. Yagi, Phys. Rev. B 82 (2010) 184117.
- [25] M. Nagumo, Fundamentals of Hydrogen Embrittlement, Springer, Singapore, 2016.
- [26] G. Benamati, E. Serra, C.H. Wu, J. Nucl. Mater. 283–287 (2000) 1033–1037.
- [27] T. Suzudo, M. Yamaguchi, A. Hasegawa, Modell. Simul. Mater. Sci. Eng. 22 (2014) 075006.
- [28] R.W. Balluffi, J. Nucl. Mater. 69&70 (1978) 240.
- [29] H. Schultz, Mater. Sci. Eng. A141 (1991) 149.